Design and Development of Nanosensor Based Soil Moisture and Temperature Monitoring System for Agriculture

Synopsis submitted in fulfilment of the requirements for the degree of

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By

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Synopsis of the Doctoral Thesis

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1. Introduction

Temperature has many different definitions and interpretations given by different organizations and experts. Some of the definitions of temperature are: World metrological Organization (WMO) defines temperature as a physical quantity characterizing the mean random motion of molecules in a physical body; Temperature is a measure of how warm or cold an object is; Temperature is related to the random thermal motion of the molecules in a substance and It is a measure of the average translational kinetic energy of molecules in a material. The main source of soil temperature is solar radiation[1]. The soil temperature ranges from -20℃ to 60℃ and may vary from place to place. The biggest changing range of soil temperature is 0 ~ 40°C[2]. The optimum average range of soil temperature for plant growth is between 20 and 30°C[3].

Among the various types of temperature measuring devices, thermistor has several potential advantages like having a higher temperature capability; does not need extension lead wires and conductors or cold-junction compensation; has high sensitivity and requires simple construction, leading to a possible low system cost [4].

Some of the challenges of soil temperature measurement are non-uniform temperature distribution, low resolution, self-heating (power dissipation) effect, the short lifespan of the sensors, high cost, high power consumption, existence of poison components and low accuracy.

The term "soil moisture content" is widely accepted as referring to the water that may be evaporated from the soil by heating to between 100 and 110ºC, but usually at 105ºC, until there is no further weight loss. This is the thermogravimetric method which is the standard reference method. As **Gardner** stated, "the choice of this particular temperature range appears not to have been based upon scientific consideration of the drying characteristics of the soil." Its origin probably has more to do with the notion of ensuring evaporation of liquid or "free" water and the relative ease with which determinations can be made by oven-drying samples.

Soil moisture determinations measure either the soil water content or the soil water potential. The water content in the soil can be directly determined using the difference in weight before and after drying a soil sample usually referred to as the gravimetric method. This method expresses gravimetric water content (θ_g, gg^{-1}) as the weight of water divided by the weight of dry soil, i.e. the ratio of the mass of water present in a sample to the mass of the soil sample after it has been oven-dried at 100-110℃ to a constant weight. The thermo-volumetric method (or simply volumetric) gives volumetric water content (θ_v, %) as the volume of water held within a volume of oven-dried undisturbed sample (soil core). Although these direct methods are accurate (±1%) and inexpensive, they are destructive, time and labour-intensive, and do not allow for replication in the same location. Alternatively, many indirect methods are available to monitor soil water content. These methods estimate soil moisture by a calibrated relationship

with some other measured variable. The suitability of each method depends on cost, accuracy, response and recovery time, installation, intended use, management, and durability.

Depending upon the quantity measured, indirect techniques are classified into volumetric or tensiometric[5] methods[6]. While the former gives volumetric soil moisture, the latter yields soil suction or water potential (i.e. tension exerted by capillarity) [7]–[14]. Heat dissipation method soil moisture sensors are important because of its wide measurement range, accuracy and low cost. Pedro Carvalhaes Dias et al [15], [16], Subir DAS et al [17], Antonio Valente et al [18] and Vinay S Palaparthy et al[19]–[22] have been working on these sensors to optimize power consumption by using efficient bulk components, decreasing the diameter of the probes and the separation distance between the probes but further decreasing the dimensions leads to easy breakage of the probes and the absence of sufficient soil between the probes. Finally, these will result in a short life span of the sensors and inaccurate measurement results. They also used one temperature sensor to detect the maximum change in temperature which is dynamic that may lead to error in measuring the exact change temperature in real-time.

There are so many challenges for the current soil moisture content measuring devices like low accuracy, low resolution, nonuniform distribution of soil moisture, high cost, high power requirement, high technical skill requirement to use the sensors, lack of technology that can manufacture optimized soil moisture sensors in any aspect, soil dependent calibration, slow response time, poor sensor installation, nonuniform distribution of soil components that affect soil moisture content, some sensors are hazardous, and low coverage area of the field.

2. Problem identification

Agriculture is the backbone of every nation of the world. The life of human being is either directly or indirectly depends on agricultural activities, particularly in the developing countries where the means of subsistence and economy has mainly relied on it. In these countries, more than 65% of the population leads their lives based on this economic sector. Agriculture practice involves farming, feedlots, grazing livestock, poultry raising, dairy farming, and aquaculture activities[23]. The use of agricultural technology improves the productivity of the agricultural sector hence enhances the lifestyle of the growing world's population. To this effect, the output of agricultural technology has to be expanded to the agriculturalists too!

The productivity of the farming sector, the type and the amount of product, are affected by natural disasters and/or anthropogenic activities. The natural hazards, particularly in developing nations, are accountable for about 22% of the total damage and losses of agricultural sectors. These include hurricanes, floods, fires, earthquakes, pests, and tornadoes[24]. They threaten agricultural production and development and often have cascading negative effects on the national economies. Selecting the improper type of seed to be planted, improper irrigation

practice, inappropriate season for planting, inappropriate land preparation for planting and misuse use of fertilizers are some of the manmade disasters of agricultural productivity.

The use of modern agricultural technologies enhances agricultural productivity [5],[6]. Practising automatic irrigation, checking soil condition before, after and during planting, selecting appropriate seed and season for plating are to mention a few of them.

For the growing of plants, three factors are most important: first the soil nutrients and soil properties; second the properties of the seeds and most important is the soil moisture level and temperature [2]. So proper handling of soil temperature and moisture is very important in agriculture to increase crop yield. The importance of soil moisture-controlling in agriculture are: automatic irrigation, water-saving [3],[4], increase product or yield [2],[4] the level of acid and pollution control[5], soil salinity control[6] and soil erosion control whereas soil temperature affects photosynthesis, respiration, transpiration, the water potential of the soil, soil translocation, and microbial activities[7]. Inappropriate soil moisture and temperature may lead to a 0% yield from a given piece of agricultural land.

Farmers and agricultural scientists have been trying to measure and estimate soil moisture content and temperature using different methods[10], [12], [27], [28]. Traditionally, farmers estimate soil moisture and temperature using their own sense organs like skin and naked eyes. They also use season and peak time to estimate soil moisture and temperature contents. In addition, farmers and soil scientists use modern instruments and devices to measure soil moisture content and temperature. They use different thermometers to estimate soil temperature[29], [30]. But still, there are many challenges to estimate soil temperature using the available thermometers. Some of the challenges are cost, power consumption, complexity of fabrication and operation, size, ease of operation, low resolution, durability, presence of poison chemicals and accuracy. There are also many numbers of existing modern methods to estimate soil moisture content. These are gravimetric methods, volumetric methods, and neutron moderation methods. Still, these methods have also been suffering from many challenges like the coverage area, durability, poison, power consumption, size, response time, recovery time and accuracy.

In this work, we have been designed and fabricated nanomaterial-based soil temperature and soil moisture meters to improve some of the challenges of the existing systems like the simplicity of operation and fabrication, poison, accuracy, cost, and power consumption.

3. Objectives

The objectives of this work are: synthesises and characterization of nanoceramic powder; preparation and characterization of electric heaters; fabrication and Calibration of the soil moisture and temperature sensors and embedded system development.

4. Thesis Organization

The report is organized into 8 chapters and the general description of each chapter is given as follows.

Chapter 1: Describes introduction which consists of background and motivation of the research, the organization of the research, soil definition, soil profile and formation, soil composition, soil classification, soil properties, soil parameters to be detected, and the role of nanotechnology in the recent agriculture in different aspects.

Chapter 2: Discusses the related literature on soil temperature and soil moisture content measurement and also states the objective of the research. It also discusses the definitions of temperature and soil moisture, the source of these parameters, factors affecting them, the need for measuring them in agriculture, and the challenges of the already existing soil temperature and moisture content measurement systems. The problem of the statements and the general and the specific objectives of the thesis are also stated here.

Chapter 3: Deals about the fabrication methods and the characterization techniques of the nanomaterials for heating and temperature sensing applications used by the researcher. It also describes the detailed specifications of spectroscopy and microscopy tools used to fabricate and characterize the nanomaterials.

Chapter 4: Explains the thermistor nanomaterial fabrication and characterization. It also discusses the fabrication, calibration, embedded system design and development, and testing of the thermistor based soil thermometer.

Chapter 5: Discusses the fabrication, characterization, and comparison of FTO and ITO based electric heaters for soil moisture estimation applications. It also explains ITO nanopowder based heater fabrication and characterization.

Chapter 6: Deals with DPHP and TPHP digital and handheld soil moisture and temperature sensor fabrication, calibration and testing.

Chapter 7: Deals about accessing soil temperature and moisture data using IoT technologies through the ThingSpeak IoT platform at a distance.

Chapter 8: Summarizes and mentions some future outlooks of the research.

List of references, appendices and notable outcomes of the research work were also included in the final part of the thesis report.

5. Nanoceramic $NiMn_2O_4$ Powder-Based Resistance Thermometer for Soil Temperature Measurement

i) Nanoceramic $NiMn_2O_4$ Powder synthesis and characterization

Materials and Equipment

Nickel (II) acetate tetrahydrate and manganese (II) acetate tetrahydrate, HNO_3, distilled water, magnetic stirrer, alumina crucible, agate mortar and pestle, FeSEM, EDS, XRD, Origin pro8, and muffle furnace.

Experiment

Step1: prepare 2gm of nickel (II) acetate tetrahydrate and 1.97gm of manganese (II) acetate tetrahydrate in a separate beaker.

Step2: dissolve each powder in 10ml distilled water with a magnetic stirrer at 80℃ on a hotplate for 2-3 hours.

Step3: mix the above two solutions together and stir for 30 minutes more.

Step4: stop stirring the solution and add some drops of HNO_3 to maintain the pH and facilitate gel formation.

Step5: dry the solution at 100℃ until the gel was formed and dried.

Step6: crush/grind the dried gel using agate mortar and pestle.

Step7: calcine the powder at 1000℃ for 3-4 hours.

Result and Discussion

The calcined powder is characterized using FeSEM/EDS for its morphology, powder size, and composition. The FeSEM result shows that the crystalline morphology of the ceramic powder and the size whereas, the EDS result confirms that the powder contains Ni, Mn, and O_2. The X-ray diffraction patterns are taken in the range of 10–80º in order to cover the most intense peaks of the $NiMn_2O_4$ phase. Nine $NiMn_2O_4$ powder diffraction peaks are found. The maximum peak is at an angle of $2\theta = 35.2922^0$. The maximum FWHM is 0.34º for the eighth peak. From the XRD powder result, using the Scherrer equation, the average crystallite size which is about 34nm is calculated. The Scherer equation is given as in equation (1).

$$D = \frac{0.9 * \lambda}{\beta * \cos(\theta)} \quad (1)$$

Where λ is the x-ray wavelength of CuKα source 0.154059nm, θ is the Bragg's angle and β is the full width at half maximum (FWHM) of the diffraction peak in radians. The XRD powder result also confirms the existence of a spinel structure of the resulting nanoceramic powder.

ii) Thermometer Design, Development and Testing

Materials and techniques

Electrically insulating but thermally conducting tube, $NiMn_2O_4$ nanoceramic powder, aluminium foil, aluminium lead wires, FTO coated glass heater, LM35 IC temperature sensor integrated with Atmega328 microcontroller, a personal computer with programming software, DC power supply, and 16X2 LCD display.

Experiment

The aluminium lead wire is inserted into an electrical insulator but thermally conductor tube followed by folded and rolled aluminium foil pieces compressed to this side of the tube. Then on the opened side of the tube 0.25gm of $NiMn_2O_4$ nanoceramic powder is pressed. Finally, this opening is closed by rolled aluminium foil pieces followed by an Aluminum lead wire. The calibration of the thermistor is made by collecting and recording 16 temperature versus resistance data from 31.28℃ to 41.06℃ temperature range with a 1 minute time interval of recording each sample. FTO coated glass heater is used as a heat source. The temperature is measured by the LM35 IC temperature sensor integrated with Atmega328 microcontroller. The resistance is measured using a digital multimeter while heating the thermometer.

Thermometer Modeling: Both Stein and Hart, and the β-parameter equation models for thermistor are used. For the Stein and Hart model, the coefficients obtained are $A = -6.489925698 * 10^{-3}$, $B = 1.014576378 * 10^{-3}$, and $C = -0.1.598860877 * 10^{-6}$. In the case of the β-parameter equation model, the values of the required parameters become: $R_0 = 1233646\Omega$ and $T_0 = 298.15K$ are taken and $\beta = 6332.36K$ is calculated. The characterization and calibration results are depicted in Figure 1.

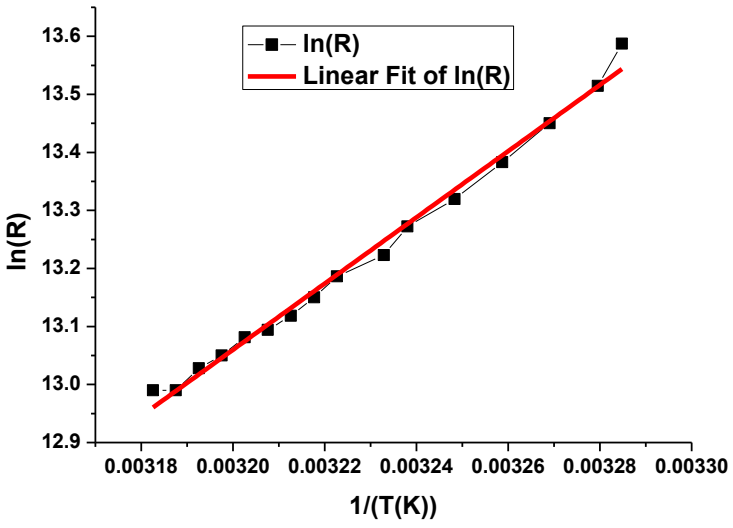

Figure 1 $1/T(K)$versus $\ln(R(\Omega))$ graph of the thermistor with a linear fit

Result and Discussion

The Stein and Hart model for the thermistor becomes:

$$^1/_{T(K)} = -6.489925698 * 10^{-3} + 1.014576378 * 10^{-3} * ln(R) - 0.1.598860877 * 10^{-6} * (ln(R))^3 \quad (2)$$

Where A $= -6.489925698 * 10^{-3}$, B $= 1.014576378 * 10^{-3}$ and

C $= -0.1.598860877 * 10^{-6}$ and the β-coefficient model becomes:

$$^1/_{T(K)} = {}^1/_{298.15\text{K}} + {}^1/_{6332.36\text{K}} * \ln(\frac{R}{123.3646\text{K}\Omega}) \quad (3)$$

Where $R_0 = 1233646\Omega$, $\beta = 6332.36$K and $T_0 = 298.15$K

The two thermistor models are compared. They are compatible with the temperature range of 27.5℃-47.5℃. The theoretical result also agrees with the experimental result. There exist a good fit between third-order polynomial (Stein and Hart model) and the resulting tested values of the thermistor with the adjacent R square of 0.99639. Therefore, the mathematical models are representative of the practically measured thermistor characteristics. This thermometer can measure the soil temperature up to 60℃. The unique features of this thermometer are the simplicity of construction, three digits (0.001) resolution, does not require complex circuitry, lead-free, low cost, easily fabricated nanomaterials based with the first time fabrication and calibration techniques, and use of economical fabrication method.

Embedded System Development: The fabricated thermistor, Atmega328 microcontroller, stainless steel cylindrical tube, Computer, 16X2 LCD display, and 10cmX10cmX4cm 3-D printed plastic box for packaging, and 5V DC power supply hardware incorporated with microcontroller programming software are used for the embedded system development. The 15cm long, 7mm inner diameter and 8mm outer diameter stainless steel tube is used as a probe to hold the thermistor. The stainless steel tube has two main functions namely protecting the sensor from physical and chemical damages and makes it be easily inserted to the ground during installation.

Thermometer Test: The fabricated thermometer has been tested by taking soil samples at a different temperature. LM35 IC temperature sensor and the fabricated thermometer are compared. The readings are almost the same except small variation after the decimal point of the reading that is the maximum error read from the results is less than 0.5℃.

6. Nanomaterial-Based Heater Fabrication and Characterization

i.Fluorine Doped Tin-Oxide (FTO) and Indium Doped Tin-Oxide (ITO) Electric Heaters

Materials and Equipment

Acetone, Deionized water, FTO coated and ITO coated glasses, silver paste, standard thermometer, digital multimeter, a hot gun, and digital ultrasonic cleaner.

Experiments

ITO Coated Glass Heater: The ITO coated glasses are square in dimension with area 2.6cmx2.6cm and 1mm thickness with the thickness of the film in the nano range. The thin film of ITO coated glass surface resistance is 70-100Ω/sq. First ITO coated glass has been cleaned. Then, silver paste from Sigma Aldrich was made at 5mm size on to two opposite ITO coated sides of the glass using the brush by masking or covering the remaining central part of the glass with tape in the open air. Two wires were connected on the two silver pasted portion of the well-prepared glass. Then to protect the heater from damage, the heater was covered with heat resistance Teflon tape.

The experimental setup to characterise the fabricated heaters consists of the 5V DC power source, LM35 integrated circuit temperature sensor embedded with Atmega328 microcontroller and serial monitor of the computer to display the temperature data. The temperature reading was recorded using the LM35 integrated circuit temperature sensor embedded with Atmega328 microcontroller for the first 10 minutes, after supplying the specified power. The records of the temperatures were done in a 1 minute time interval.

FTO Coated Glass Heater: FTO coated glass is also a square in dimension with the same area of the ITO coated glass with the thickness of 0.2cm. FTO coated glass was cleaned as an ITO coated glass. Silver paste from Sigma Aldrich was made as ITO coated glass. Two wires were connected on the two silver pasted portions of the well-prepared glass. Then to protect the heater from damage, it was covered with heat resistance Teflon tape. The experiment to characterize the fabricated heaters is the same as that of ITO coated glass heater.

Results and discussion

For the 5V DC power supply, the FTO coated glass heater attains its steady-state temperature after 10minutes, and the maximum value becomes 71.87℃. This process is repeated three times in the open air. Actually, the heater started attaining its steady-state temperature after 9 minutes. In the first a 1 minute of time, the heating speed is slow in all the three repeats. This is because, in the first few minutes of the heating process, the heat energy

of the heater should be equilibrated with the ambient energy. But for the next 4 minutes of the heating time, the heating speed is increasing almost linearly. This is because of the ambient heat and the heater's heat is only in a small difference. From 4 to 9 minutes time, the temperature of the FTO coated glass heater increased slowly. This is because of approaching its steady-state temperature at this particular condition. After the 9^{th} minute of time, the heater started to attain its steady-state to a particular temperature of about 70℃. All those heating events are almost the same for all three cases. These small differences are due to ambient conditions. Some of the ambient conditions are the variation of humidity, temperature, and pressure. The three cases were to check the repeatability and stability of the heater. The maximum change of temperature obtained from the FTO coated glass heater becomes 39.12℃.

During cooling, in the first 7 minutes of time after switching off the electric power supply, the temperature decreases rapidly and almost linearly. This is because of large heat exchange within the ambient and also characteristics of the material. The heater takes about 15 minutes, to attain its ambient temperature. The cooling speed is slower than the heating speed. In the cooling process, the heater's activity is almost the same for the three cases except for very little variations as in the case of heating because of ambient conditions.

In the case of ITO coated glass heater, the maximum temperature recorded is 45.45℃. For the first three minutes of time, the temperature increases rapidly and almost linearly in all three cases. The reason behind this is the same as that of the FTO coated glass heater. Then in the next 4 minutes time, the speed decreased but almost the same for the three cases. After 7 minutes, of supplying power, the heater attained its steady-state temperature i.e., about 45℃. As mentioned above in the case of an FTO coated glass heater, there are ambient factors that affect the temperature of the ITO coated glass heater. The maximum change in temperature for this heater is 13.19℃. The cooling rate is very fast and almost linear in the first 3 minutes of time after the power is switched off. This time is faster than the cooling time of the FTO coated glass heater. The reason behind the variation of the cooling rate is the same as that of FTO coated glass as specified above. In the next 3.5 minutes, the cooling speed somewhat decreases but after 7 minutes later the heater attains its ambient temperature.

In general, when we compare the two heaters, FTO coated glass heater has a larger change of temperature. The time to achieve this temperature is actually longer than the ITO coated glass heater. The cooling time is also longer than the ITO coated glass heater. These might be due to the nature and thickness of the two films. One of the peculiar features of these kinds of heaters is their transparency. Transparent heaters widely used in aircraft windows, and doors of long buildings and luxuries cars used for the antifogging purpose. Again these kinds of heaters are based on thin films which are 2-dimensional nanomaterials that have many potential applications in the current and future technologies. One of the potential applications of

nonmaterial based heaters is their low energy consumption. Nano and microheaters have been widely used in gas sensors [31], [32].

ii) ITO Nanopowder Based Electric Heater

Materials

ITO nanopowder, aluminium lead wires and foil, metal cylindrical tube, Teflon tape, powder XRD, digital multimeter, DC power supply and FTO coated glass electric heater.

Experiment

1.6gm of ITO powder is measured using mini analytical balance and pressed well into the 1cm length, 1.2mm inner diameter and 0.3mm thickness Teflon tape insulated and one side blocked with an aluminium sheet and electrode metallic tube. The powder was pressed well. Then the other remaining end of the tube was blocked using folded and rolled aluminium foil and rode. The folded and rolled aluminium foil was used as a metal contact between the powder and the aluminium electrode. The electrodes used were aluminium rods. The average size of the ITO powders was determined using XRD data with the help of the Scherrer equation.

Characterization

***Temperature and Time versus Resistance Characterization*:** The effect of time and temperature on the ITO nanopowder based electric heater has been studied to check the stability of the heater. The experiments have been repeated three times. From temperature versus resistance characteristics of the heater, there exists a linear fit equation with a slope of -0.01428 and standard error of 0.01582 with adjacent R square of -0.02104. These results proved that the resistance of the ITO powder heater is independent of temperature for the given temperature range (32-39℃). In addition, time versus resistance linear fit equation with slope =-2.3360E-4 and standard error =5.21058E-4 with the adjacent R square =-0.09743 has been obtained. These results also confirm that the resistance of nano ITO powder-based heater is independent of time during operation. Therefore, the nano ITO powder-based heater is stable in its operation.

Time versus Temperature Characterization: Time versus temperature characterization is conducted by supplying 5V DC power across the heater terminals and measuring the temperature of the heater. The experiment was repeated three times. The cooling characteristic of the heater from $45.45℃ - 34.70℃$ was also studied in three repeats.

Results and Discussion

The ITO nanopowder based electric heater has been fabricated successfully using a simple (fill and press) and economical technique. Some other peculiar features of this heater are it does

not require insulation, the simplicity of operation and fabrication, lower power consumption, durable and cheap fabrication cost. XRD pattern is used to calculate the average size (38.7nm) of the nanopowder with the help of the Scherrer equation. The temperature range of the heater under the supply of 5V for about 10 minutes time was from 34.7℃-78.69℃ in ambient condition. The heater took 8 minutes time to attain its steady-state temperature. The change in the temperature became 43.99℃ higher than the thin-film heaters described in the previous section. The increase in temperature has some variations because of the ambient conditions like temperature and humidity. The cooling process of the heater from 45.45℃-34.70℃ was taken place within about 3minutes of time. The resistance of the heater is almost not affected by temperature and time. Comparison of the three fabricated electric heaters in terms of steady-state temperature, maximum change in temperature, steady-state attaining time and recovery time are shown in Table 1.

Table 1 Comparison of the three electric heaters

No.	Heaters	Max.temp.(℃)	Δ.temp.(℃)	Steady state Time(m)	Recovery Time(m)
1	ITO film	45.45	13.19	7	7
2	FTO film	71.87	39.12	10	15
3	ITO powder	78.69	43.99	8	3

7. Nanomaterial-Based Soil Moisture Sensor Fabrication and Testing

i) Application of FTO Coated Heater in DPHP Based Volumetric Soil Moisture Content Estimation

Materials and Equipment

Self-fabricated FTO coated heater, LM-35 temperature sensor, Microcontroller with the associated software, sample holder and soil samples.

Experiments

The heater was covered with Teflon tape to resist soil and water contamination during installation, the sample holder is a long plastic cylinder with a 1.5mm diameter and 6mm height. Four different compositions of laterite soil samples were collected from the Joint Director Agriculture office, D.K.District, Mangalore. These four soil samples are different in pH, electrical conductivity (EC), Potash (Po), phosphorus (P) and organic carbon (O.C) contents. They are designated as soil samples a, b, c and d.

Sample preparation: The $5cm^3$ of dry soil sample was taken and 0 to 6.5ml of tap water was added to the first 1.5ml water content then the next samples were 0.5ml additional water difference each. The mixture was mixed very well. Then the prepared soil sample was placed properly on the sample holder. Then it was placed on the FTO coated glass heater at a steady-state temperature. After 4 minutes of time, the temperature was recorded each and every fraction of samples.

Soil Moisture Detection: The fabricated FTO coated glass heater was used as the heat source of the dual probe heat pulse (DPHP) soil moisture content estimation method. LM35 IC temperature sensor embedded with Atmega328 microcontroller was used as the temperature sensor. The height of the sample holder was about 6mm. so that the temperature sensor and the heater were separated by 6mm distance. 5V DC electric power source was supplied to the heater. The electric power consumed by the heater was found to be $5v * 2.13mA = 10.65mW$. First, the four dry soil samples a, b, c, and d were tested respectively. The temperature readings of the dry soil samples were 37.63℃, 36.66℃, 51℃ and 37.63℃ respectively. The experiment was repeated three times for each sample to check the repeatability of the sensor. From the result, the dry soil thermal conductivity is greater than that of moist soil in all the three samples. This means the specific heat capacity of water in the soil is greater than all other elements of the soil. There is also a small error in the repetition of the measurements. These errors might be due to environmental factors. The experimental results are shown in Figure 2.

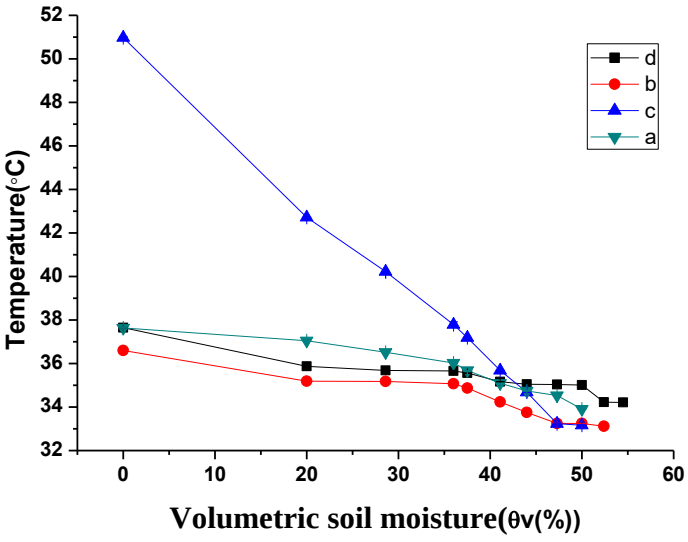

Figure 2 Soil water content versus temperature graph for the four soil samples

Volumetric moisture content versus temperature results of the four samples are different. This is because DPHP soil moisture content measurement method depends on the soil composition, the maximum change in temperature, the separation distance between the heater and the temperature sensor probes and the heat energy supplied by the heater to the soil samples.

There is a strong correlation between the change in temperature and the volumetric water content of the given soil samples. Soil sample c shows the best linear fit of all.

Some of the advantages of these kinds of heaters in DPHP soil moisture content estimation method are durability and small power consumption. These heaters are durable in soil moisture content measurement because of their durable glass substrate based heater and good adhesion between the film and the substrate. The electric power used in this experiment was 10.65mW. Therefore there is a possibility of using nanomaterial-based heaters in heat pulse method soil moisture content estimation somewhat accurately, efficiently and for a long period of time. One of the major problems of these kinds of heaters is non-uniformity of temperature over the surface of the film. But the uniformity of the temperature is not a big issue in DPHP method soil moisture content estimation.

ii) Triple Probe Heat Pulse (TPHP) Method Volumetric Soil Moisture Content and Temperature Measurement

Materials/Equipment and Methods

Two $NiMn_2O_4$ nanoceramic powder-based thermistors, ITO nanopowder based electric heater, soil samples, dual probe heat pulse (DPHP) method which is modified to the TPHP method is used for soil moisture content estimation.

Experiment

In the probe design, three 15cm effective lengths and 6mm inner diameter and 1mm thick stainless steel tubes are used. Two thermistors with a room temperature resistance of 123KΩ and 5.6MΩ and β value of 6332K and 5489K respectively and one ITO based electric heater were taken and kept inside the stainless steel tubes about 2cm distance away from the tip. One thermistor is used to measure the temperature of the heat pulsed soil at a distance of 6mm from the heat source and the other one is used to measure the reference temperature and soil temperature continuously. It is placed 28mm away from the heat source. This distance is chosen to avoid the effect of the heater on this thermistor[33]. For the electrical design, we have used Atmega328 microcontroller, simple resistance to voltage converter electric circuit, 16x2 LCD display, breadboard, copper wires and 10cmx10cmx4cm 3-D printed plastic box for packing.

During the calibration of the TPHP soil moisture estimation meter, the following parameters have been used: x_m=0.45, x_o=0.05, Pulse duration = 10m, r=6mm, heater length=1m, V =5v and R_{ITO}=6.97Ω. Finally, the following calibration equation (equation (4)) is obtained for the given soil sample.

$$\theta_v = \frac{1}{4.18}\left(\frac{7}{\Delta T} - 0.989\right) \quad (4)$$

A laterite soil which is common agricultural soil in D.K. District with the composition and properties of $(PH) = 7.08$, Electrical conductivity $(EC) = 0.070A$, Phosphorous$(P) = 21.62Kg/H$, Potash$(Po) = 29.12Kg/H$, Organic Carbon$(OC) = 1.45Kg/H)$ from Joint Director Agriculture office, D.K.District, Mangalore is taken and different moisture-related experiments are performed. **Embedded System Development and Field Testing:** The system consists of hardware and software components. The hardware components are an Atmega328 microcontroller, two self-fabricated thermistors as temperature sensors, small breadboard (to develop the signal conditioning and the LCD interfacing electronic circuits), 16x2 LCD display, and 10cmx10cmx4cm 3-D printed plastic box for packaging. After completing the electric circuit, we packed it in 3-D printed plastic box. The overall mass of the device is about 125gm which is light for transportation in the case of the handheld in-situ measurement.

Results and Discussion

Measurement of volumetric soil moisture content and soil temperature for the given particular type of soil with a different amount of soil moisture content and temperature is studied in detail using the fabricated nanomaterial-based TPHP sensor.

In the case of dry soil for the first 5minutes of time, the temperature of the soil increases rapidly. This is because, in the beginning, there is a high heat energy difference between the heater and the surrounding soil. As a result of this, the heat is transferred rapidly mainly by conduction[34], [35] and the temperature increases within a short time duration, then after, the soil attains its steady-state temperature. This means the heater energy becomes equilibrium with the surrounding soil heat energy. As a result of this, heat flow from the heater to the surrounding stops.

The same soil sample has been taken and the time versus change in temperature characteristics of two different volumetric soil moisture contents are studied. The volumetric soil moisture contents of the sample soil are 0% and 23%. When the soil moisture content increases, the change in temperature decreases and vice versa. This result is also valid for decreasing soil temperature which shows a slow decrease in temperature for larger moisture content.

Again, we took the soil sample with a soil moisture content of 0%, 23%, and 28% and studied their relationship with temperature. From these volumetric soil moisture contents of the soil sample, we have got a similar result which is as volumetric soil moisture content increases, the temperature decreases. The reason behind this is water has the highest volumetric heat capacity of all other soil components that is 4.18MJ/m^3°C as specified in [35]. This result is

true for all other soil types with specific calibration and shows the validity of the already existing model as shown from Figure 3-Figure 5.

For the given particular type of soil sample, in the heater temperature range of 31℃-60℃, the total rise in the temperature of sensor (ΔT) was found to be between 1.36℃ and 7℃ in measuring volumetric soil moisture content of the soil in the range of 0-100% as explained in [17], [36].

The thermometer can measure the soil temperature up to 50℃ which covers the biggest changing range of soil temperature which is 0 ~ 40 ℃ as mentioned in[2] and the optimum average range of soil temperature for plant growth is between 20 and 30℃ as mentioned in[3]. The particular feature of this thermometer is it can read soil temperature in two or three-digit precision.

The laboratory tested volumetric soil moisture content versus the fabricated TPHP method tested soil moisture content for the given soil sample have been compared. The corresponding moisture contents are almost equal and the slope becomes approximately 1 with adjacent R square of 0.99178. The result is depicted in Figure 6. The average relative response shows about 101.1%. The results on our system are very slightly higher than the standard method. There is a good linear fit between the volumetric soil moisture and the change in temperature with the adjacent R square of 0.97535 as shown in Figure 7. This result confirmed the existence of an inverse linear relationship between soil moisture content and a change in temperature.

The TPHP has been designed, fabricated and tested in the field. The percentage difference between gravimetric based and TPHP method soil moisture content measurement is less than 2.8%. The average sensitivity of the sensor becomes 2.6°C per unit change (m^3m^{-3}) in water content. The heater consumes 0.515W electric power and the embedded system need only 5V DC power supply.

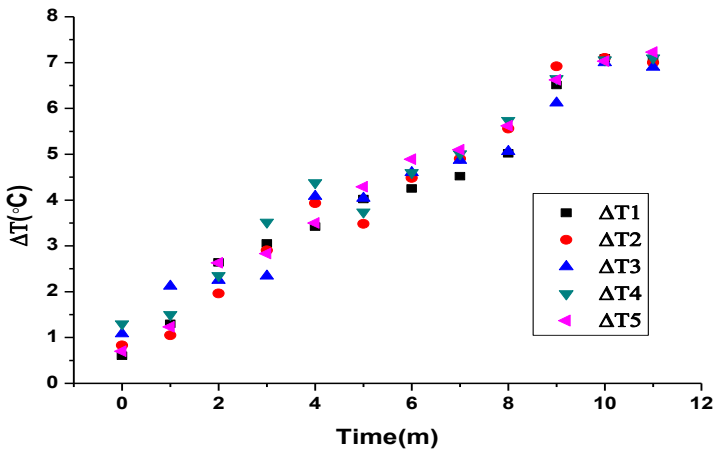

Figure 3 Time versus change in temperature graph for dry soil sample repeated 5 times

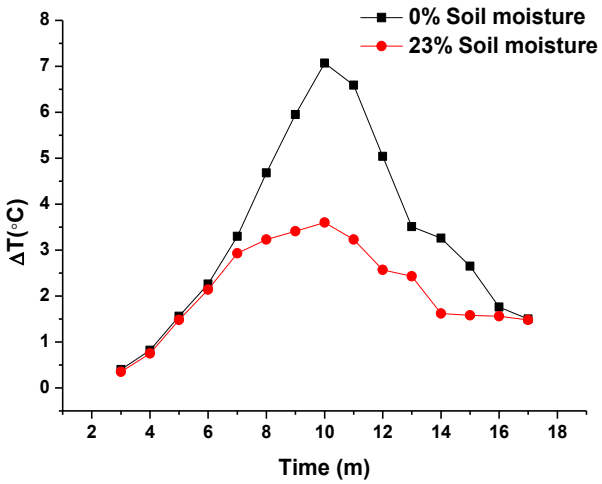

Figure 4 Time versus change in the temperature graph

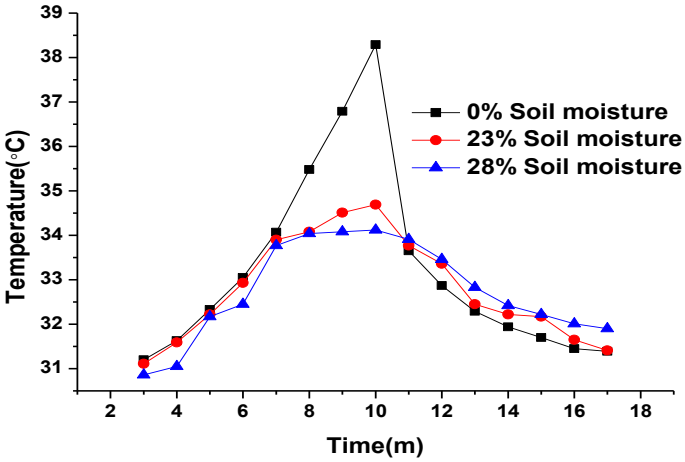

Figure 5 Time versus temperature graph for the three soil moisture samples

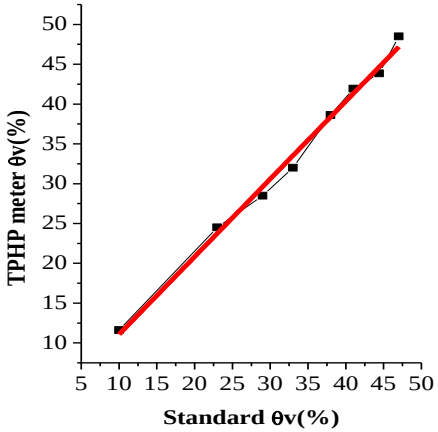

Figure 6 laboratory tested soil volumetric moisture in % versus TPHP sensor tested volumetric moisture in %

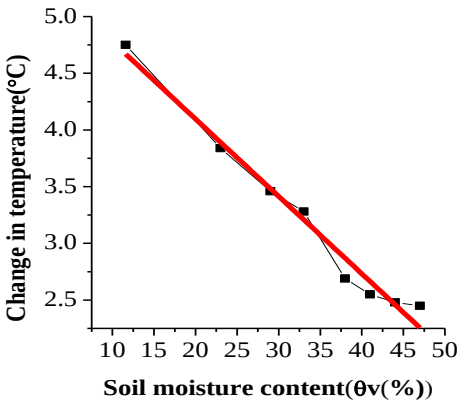

Figure 7 θ_v versus ΔT graph

8. Real-Time Monitoring of Soil Moisture and Temperature Using IoT Technology

Materials

NodeMCU development kit, Nanoceramic based thermistor, ITO Nanopowder (32nm) based electric heater, computer, two 15cm height stainless steel cylindrical tube, 3D printed plastic package, Arduino IDE and ThingSpeak IoT platform

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Experiment

Design and prototype of the system

Hardware design*:* We have used a self-fabricated $NiMn_2O_4$ nanoceramic powder-based thermistor with a room temperature resistance of ($R_{tho} = 5M\Omega$) and ITO nanopowder based heater with ($R = 24\Omega$). The dimensions of the cylindrical stainless steels are($l = 15cm$, inner diameter $= 6mm$ and outer diameter $= 8mm$). We have used the DPHP/TPHP soil moisture sensor so that the heater and the temperature sensor inside the stainless steel cylindrical tube are kept $6mm$ away from each other. The heater and the thermistor were kept in 1cm distance away from the pointed tip of the stainless steel tubes. In the signal conditioning circuit, a $5M\Omega$ resistor was connected in series with the thermistor to get voltage signal as an input for the NodeMCU development kit. The main purpose of the signal conditioning circuit is to convert the resistance of the thermistor into a voltage signal which is a suitable input signal for NodeMCU development kit.

Software design: The software part of this work can be classified into three main sections. These are the initialization section, the setup function section and the loop function section. The initialization section initializes write apikey, network elements (password and user ID), channel number, server name, all the thermistor constants and power supply. It also declares a WiFi client. The setup function section has mainly two tasks. These are initializing the serial communication, and making and checking the Wi-Fi connection. The last section of the software evaluates the soil parameters analysis that are temperature and moisture, displays all the analyzed values to the serial monitor, pushes the data to the channels, displays the data in the ThingSpeak dashboard, determines the time duration for updating the new data to the channels and updates the data on channels.

Results and Discussion

The sensed soil moisture and temperature data are collected, processed and transmitted to ThingSpeak application and API software using NodeMCU development kit and displayed through the ThingSpeak channel in the form of a graph. ThingSpeak is an open-source Internet of Things (IoT) application and application programming interface (API) to store and retrieve data from things using the hypertext transfer protocol(HTTP) over the Internet or via a Local Area Network(LAN) that enables to collect, store, analyze, visualize, and act on resulted data from sensors or actuators manually or automatically. The results of ThingSpeak can be accessed by private or public channel users' account. The private channel account displays data only to the authorized person but the public channel can display the resulting data form of a graph to the public.

In this work, we report the soil temperature using Celsius and Fahrenheit scale. The data show that the soil temperature is 21℃ at a particular instant of time and then increased to 26℃. The volumetric soil moisture content is 3% at some particular point of time and finally, it stabilizes at 31%. The system also shows the location of the sensor by inserting the location of the place where the sensor is installed in the channel. The location field in the channel requires the longitude and the latitude values of that particular place of which sensors are installed. The sensor system was tested by taking a pot plant in the department of electronics, Mangalore University.

9. Summary

In this work, nanomaterial-based thermometer, dual probe heat pulse (DPHP) and triple probe heat pulse (TPHP) sensors for soil temperature and volumetric moisture content testing have been designed, fabricated, characterized, calibrated and finally tested in the field (pot plant).

Thermometer from NTC nanoceramic thermistor powders is fabricated easily using a simple cost-effective method called fill and press method and procedure. The procedures are synthesizing nanoceramic powders, characterizing the powders, fabricate the thermistor, calibrate the thermistor, pack it and finally test the thermometer in the field. The unique features of this thermometer are: two or three-digit precision temperature measurement and does not require any pellet making or film-making process. One of the important advantages of this thermistor is that it is lead-free and this is the unique kind of thermometer that is based on nanomaterial for this particular application.

For the fabrication of soil moisture meter, heat energy is needed. Heat energy is one form of energy that can be generated and transformed from different sources in many different ways. One method of obtaining heat energy is converting electrical energy to heat energy using energy converters. One simple energy converter is modified Tin Oxide (TO) coated glass. Two modified tin oxide (TO) based glasses have been taken and constructed, characterized and compared as electric heaters. FTO coated glass heater is constructed after cleaning, pasting with silver paste and connected with lead wires. After that 5V DC electric power source is supplied and the resulting temperature has been recorded for some time, in a 1-minute interval. The temperature measurement has been done using an automatic home assembled LM35 IC temperature sensor embedded with Atmega328 microcontroller. The reading showed that the maximum record became 71.87℃ within 10 minutes after supplying the power. The maximum change of temperature for this sample is 39.12℃. The FTO coated glass heater takes about 15 minutes to attain its ambient temperature. This showed that the cooling speed is slower than the heating speed and also that of ITO based heater.

ITO coated glass heater has been constructed and characterized as an FTO coated glass heater and finally compared to an FTO coated glass heater. The maximum temperature recorded for this heater is 45.45℃. The maximum change in temperature for this heater has been recorded as 13.19℃. This is attained in the first 7 minutes of time which is faster than the FTO coated glass heater. The variations of the temperature recording for both heating and cooling the samples in all 3 rounds of the experiment are due to ambient variables. The change in temperature, steady-state attaining time, and cooling durations of the two specified samples are due to differences in the thickness of the films, and nature of the materials.

Low power and cost-effective electric heaters have also been fabricated using the cost-effective technique from nanomaterials. Here, ITO nanopowder is pressed in the insulated metallic cylindrical tube and lead wires are extended from the two ends based electric heater is fabricated for soil moisture content detection application.

FTO thin film coated glass electric heater based DPHP volumetric soil moisture content and temperature sensor has been designed, fabricated and tested. The incorporation of film heaters is advantageous over the usually wired heaters because of their durability and low power

consumption. In addition, film heaters are simple and easy to incorporate into the DPHP sensor system.

TPHP meter for soil moisture and temperature measurement application has also been designed and fabricated from the fabricated nanomaterial-based heaters and temperature sensors successfully for the first time and then calibrated and tested. The meter has been tested in the pot plant and works accurately with the percentage error less than 2.8% and the gravimetric(standard) and the TPHP sensor moisture contents are almost equal and the slope of the linear fit equation becomes approximately 1 with adjacent R square of 0.99178. The heater consumes 0.515W electric power and the electronic circuit requires 5V DC power supply. The meter can display or provide two or three-digit precision output for both temperature and moisture results. Some of the advantages of this sensor are low cost, less weight, and simple to construct and manipulate.

IoT technology-based data accessing is also implemented using the fabricated temperature and moisture sensor on the ThingSpeak open-source IoT platform.

10. Notable outcomes of the research work

List of Journal papers published

1. **Thin film Dual Probe Heat Pulse (DPHP) Micro Heater Network for Soil Moisture Content Estimation in Smart Agriculture**
   Journal of the Electrochemical Society, 166 (2) B63-B67 (2019)
   Almaw Ayele Aniley, Naveen Kumar S. K., Akshaya Kumar A., Renny Edwin Fernandez, Shekhar Bhansali
2. **Nanoceramic $NiMn_2O_4$ Powder-Based Resistance Thermometer for Soil Temperature**
   ECS Transactions, 88 (1) 455-470 (2019)
   Naveen Kumar S. K., **Almaw Ayele Aniley**, Akshaya Kumar A., Renny Edwin Fernandez, Shekhar Bhansali
3. **Fabrication, characterization and comparison of Nanocrystalline $NiMn_2O_4$ and $NiZn_{0.2}Mn_{1.8}O_4$ NTCR Ceramic Thermistor Powders**
   J. Nanosci. Tech. 5(1) (2019) 603–606
   Almaw Ayele Aniley, Naveen Kumar S.K., Akshaya Kumar A
4. **Soil Temperature Sensors in Agriculture and the role of Nanomaterials in Temperature Sensors Preparation**
   International Journal of Engineering and Manufacturing Science. ISSN 2249-3115 Volume 7, Number 2 (2017), pp. 363-372
   Almaw Ayele Aniley, Naveen Kumar S.K., Akshaya Kumar A

5. **Hydrothermal Growth of Zinc Oxide (ZnO) Nanorods and Structural and Optical Studies for Soil pH Measurement Applications**
   ECS Transactions, 88 (1) 437-447 (2019)
   Naveen Kumar S. K., Akshaya Kumar A., **Almaw Ayele Aniley**, Renny Edwin Fernandez, Shekhar Bhansali
6. **Soil Moisture Sensors in Agriculture and The Possible Application of Nanomaterials in Soil Moisture Sensors Fabrication**
   International Journal of Advanced Engineering Research and Technology (IJAERT). ISSN No.: 2348 – 8190 Volume 6 Issue 1, January 2018
   Almaw Ayele Aniley, Naveen Kumar S.K., Akshaya Kumar A
7. **Advanced Sensor Materials Based Real-Time Soil Moisture Content and Temperature Monitoring Using IoT Technology in Smart Agriculture**
   Indian journal of environmental protection (IJEP) 39(7) 639-644(2019)
   Almaw Ayele Aniley, Naveen Kumar S.K., Akshaya Kumar A
8. **Hydrothermal Growth of Zinc Oxide (ZnO) Nanorods (NRs) on Screen Printed IDEs for pH Measurement Application**
   Journal of the Electrochemical Society, 166 (9) B3264-B3270 (2019)
   Akshaya Kumar A., Naveen Kumar S. K., **Almaw Ayele Aniley**, Renny Edwin Fernandez, Shekhar Bhansali

List of papers presented at international and national level conferences

1. Dual Probe Heat Pulse(DPHP) Method Volumetric Soil Moisture Content Estimation Using Nanomaterial-Based Thermistor and Fluorine Doped Tin Oxide Thin Film Electric Heater
   Third International Conference on Nanomaterials: Synthesis, Characterization, and Applications (ICN 2018) on 11, 12 and 13 May 2018 at Mahatma Gandhi University, Kottayam, India.
2. ITO Nanopowder Based Electric Heater Fabrication and Characterization
   Third International Conference on Nanomaterials: Synthesis, Characterization, and Applications (ICN 2018) on 11, 12 and 13 May 2018 at Mahatma Gandhi University, Kottayam, India.
3. Nanocrystalline $CaTio_3$ Ceramic Powder Preparation Using Mechanochemical Method Followed By Successive Heat Treatments
   National *Conference* on Advances in the Semiconductor Materials and Device Technologies (NCASMDT-2018) on 12-13 Feb *2018 at Jain University* Department of Physics, *Bangalore, India*

4. Nanoceramic Powder Based Thermometer for Basal Body Temperature Monitoring System

   Third International Conference on Nanomaterials: Synthesis, Characterization, and Applications (ICN 2018) on 11, 12 and 13 May 2018 at Mahatma Gandhi University, Kottayam, India.

5. Nanoceramic $NiMn_2O_4$ Powder-Based Resistance Thermometer for Soil Temperature Measurement Application

   First International Conference on 4D Materials and Systems (August 26-30, 2018) at Yamagata University, Yonezawa, Japan.

6. Advanced Sensors Based Real-Time Soil Moisture and Temperature Monitoring Using IoT Technology in Agriculture

   FIRST INTERNATIONAL CONFERENCE ON MATERIALS AND SPECTROSCOPY (ICMS-2018) 11-12 December 2018 at SAVEETHA ENGINEERING COLLEGE, Chennai, Tamil Nadu, India

7. Hydrothermal Growth of Zinc Oxide (ZnO) Nanorods and Structural and Optical Studies for Soil pH Measurement Applications

   First International Conference on 4D Materials and Systems (August 26-30, 2018) at Yamagata University, Yonezawa, Japan.

8. Fabrication of Interdigitated Electrodes (IDEs) by Screen Printing Technology and their Structural Studies

   Third International Conference on Nanomaterials: Synthesis, Characterization, and Applications (ICN 2018) on 11, 12 and 13 May 2018 at Mahatma Gandhi University, Kottayam, India.

9. STRUCTURAL AND OPTICAL PROPERTIES OF ZINC OXIDE NANOFLOWERS SYNTHESIZED USING SOL-GEL METHOD

   National *Conference* on Advances in the Semiconductor Materials and Device Technologies (NCASMDT-2018) on 12-13 Feb *2018 at Jain University* Department of Physics, *Bangalore, India*

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